MICROSTRUCTURAL CHANGES OF AISI 316L DUE TO STRUCTURAL SENSITIZATION AND ITS INFLUENCE ON THE FATIGUE PROPERTIES

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Resume

Mechanical and fatigue properties of material are dependent on its microstructure. The microstructure of AISI 316L stainless steel commonly used for the production of medical tools, equipment and implants and during the operation they can be exposed to elevated temperatures. Microstructural changes and fatigue properties of AISI 316L stainless steel due to the heat treatment consisted of annealing at the temperature of 815°C with the dwell time of 500 hours were analyzed in the present paper. Precipitation of intermetallic phases and carbides was observed as a response of the material to the applied heat treatment. Negative influence was on the fatigue strength was observed in the case of region bellow N = 10^5 cycles; however the fatigue limit remains unchanged despite to the structural sensitization.

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1. Introduction

Stainless steels are characteristic by high corrosion resistance, strength, microstructure stability and ductility [1]. They can be divided into four groups. Ferritic stainless steels are used mainly for electrical applications, martensitic stainless steels in applications requiring high strength, austenitic stainless steels for applications requiring good corrosion resistance, weldability and good forging properties, and so called duplex stainless steels with combination of previous described microstructures [2]. Most of the stainless steels can be used for surgical tools and equipment and temporary or permanent implant production [1]. Most of the medical tools and equipment produced from stainless steels used in medical industry have to be resistant to decontamination and sterilization because during these processes strong chemical detergents and hot steam or high temperatures heating are adopted [3].

AISI 316L stainless steel is one of the most commonly used metallic materials in medicine. AISI 316L has very good corrosion resistance in human liquid (salt solution) environments [4, 5]. The corrosion resistance can be even improved by addition of Mo or Cu. Mo improves its resistance against the acids and pitting corrosion. AISI 316L steel has good weldability without need of additional heat treatment, very good formability and it is possible to polish it to the mirror like surface [6]. AISI 316L microstructure consists of austenite, what means that chemical composition of these grades has to be optimized to maintain austenitic phase stability at room temperature as well as shifting of the martensitic transformation temperature well below room temperature, what is result of the Ni addition [3].

The AISI 316L stainless steels have not toxic reaction with surroundings tissues and they are widely used in traumatological temporary devices such as fracture plates, screws and hip nails among others, owing to their relatively low cost, availability and easy processing [4, 5].

Due to the chemical composition, when the phase transformations are limited, the austenitic stainless steels are sensitive to the grain coarsening at elevated temperatures. As a result of heat treatment creation of ineligible intermetallic phases occurs. Mainly $(CrFe)_{23}C_6$ carbides are created on the grain boundaries due to the exposition of the material to the temperatures in the range from 500 to 800 °C. As a result of carbides presence on the grain boundaries the ductility, toughness and the corrosion resistance of the grain boundaries decrease [7]. The negative influence of the structural sensitization on the materials properties was proved by the evaluation of the corrosion resistance of AISI 316L stainless steel in a basic state and after sensitization in simulated human liquid (physiological solution – 0.9 % solution of NaCl) at various temperatures in [4].

In the present work is analyzed the influence of annealing at the temperature of 815 °C with the dwell time of 500 hours on the structural changes and its influence of the fatigue properties of AISI 316L stainless steel.

2. Experimental material and methodology

AISI 316L austenitic stainless steel was used as the experimental material. The materials was delivered in the shape of bars with the length of 3 000 mm and the diameter of 14 mm. The chemical composition of the experimental material, given by the producer, is shown in the Table 1.

Heat treatment was performed with the aim of structural sensitization. The performed heat treatment consisted of annealing at the temperature of 815 °C with the dwell time of 500 hours.

Rotating bending fatigue tests (R = -1) were performed at the frequency of 40 Hz (2 400 rpm) at the laboratory air at the temperature of 20 ± 10 °C. Eleven testing bars form the basic material and ten testing bars form the material after heat treatment were used for the fatigue behavior determination. The geometry of the testing bars is shown in Fig. 1.

Table 1

Chemical composition of AISI 316L stainless steel (in wt. %).

Cr	Ni	Mo	Mn	Ti	C	Si	P	S	Fe
17.32	13.68	2.73	1.89	0.002	0.026	0.65	0.028	0.026	rest

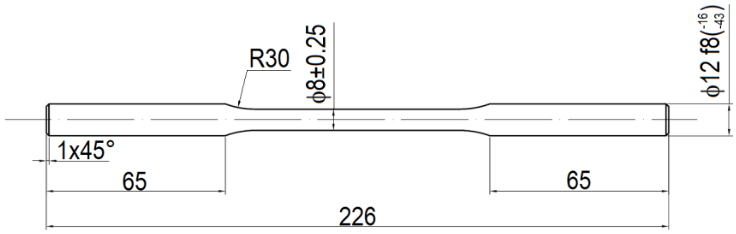


Fig. 1. Geometry of the testing bars used for the fatigue tests.

3. Results and discussion

In the microstructure of the studied AISI 316L in the basic state were observed carbides of $M_{23}C_6$ type on the not etched metallographic specimens, Fig. 2a, b. The present carbides occurred in lines due to the rolling process used for the material production. The carbide lines were created by small discrete particles lying in lines with different density.

Microstructural observation was performed on Zeiss Axio Imager Z1m Light Optical Microscope (LM). Metallographic specimens were prepared by standard procedure consisted form grinding, polishing and etching of the specimens by etchant consisted of 10 ml HNO3 + 30 ml HCl and 30 ml glycerine. Fracture surfaces were observed using TESCAN VEGA LMU II Scanning Electron Microscope (SEM).

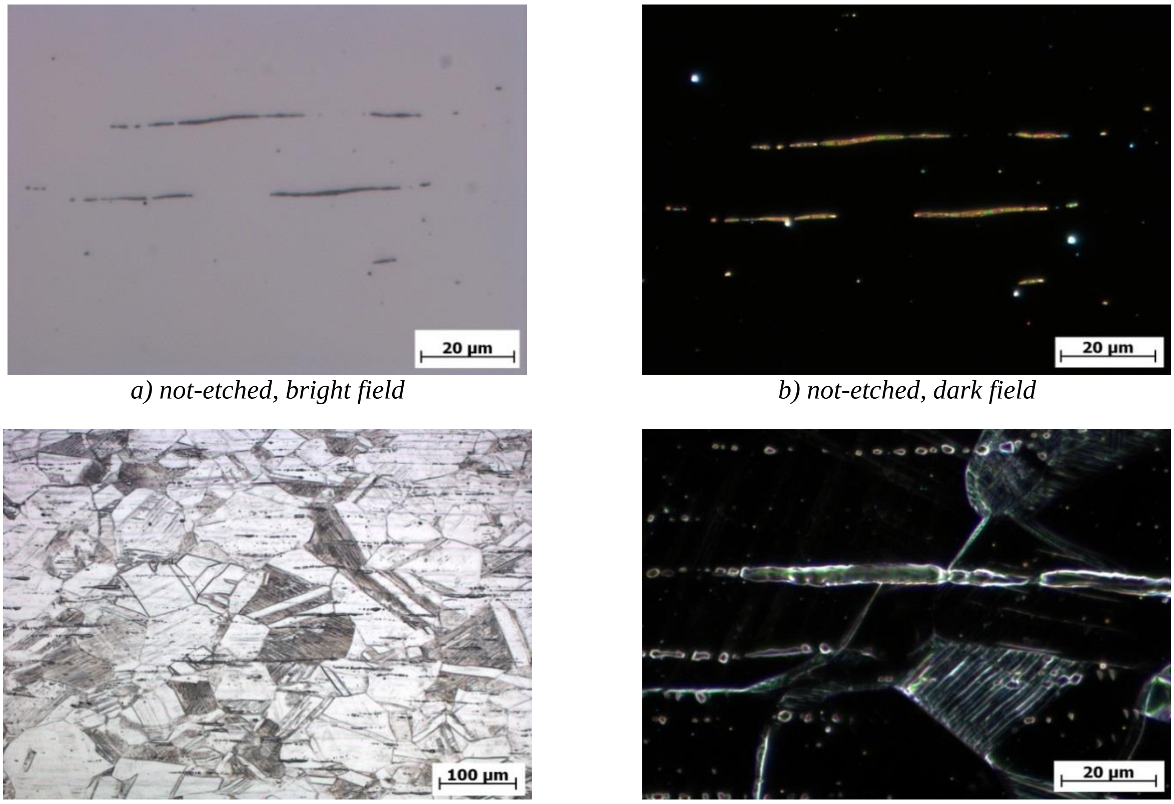
The present carbides occurred in lines due to the rolling process used for the material production. The carbide lines were created by small discrete particles lying in lines with different density. In general, the microstructure of the basic material is created by polyedric grains of austenite and rests of untransformed δ ferrite, Fig. 2c, d. Deformation twins, typical for materials with fcc crystallographic package and low stacking fault energy, were observed in the polyedric grains. The grains of δ ferrite were deformed in the rolling direction, Fig. 2c. The δ ferrite grains were present in the structure due to the production technology and they can remain in the structure even after heat-mechanical treatment [8, 9].

Also the microstructure after heat treatment was consisted of polyedric grains, Fig. 3 c. However the microstructure was significantly changed due to the applied heat treatment. The carbide lines, observed in the steel after heat treatment were created from disperse carbides, Fig. 3a, b. Due to the exposition of the material to the temperature of 815 °C for 500 hours two important microstructural changes occurred. The δ ferrite grains present in the basic material were disintegrated and large amount of Cr carbides was created. The first intermetallic phase created during heat treatment is $M_{23}C_6$, where M means elements as Cr, Fe, Mo and Ni. On the beginning the Fe is used, while later the Cr and Mo are involved [10, 11]. In the present work the carbides were observed mainly on the grain boundaries and on the twin boundaries, Fig. 3 d, which is in agreement with [10, 11] where the grain boundaries were observed as preferential precipitation places. Following preferential places for the carbide precipitation mentioned in the literature are twin boundaries and dislocations [10, 11]. The second important change observed in the present study was χ and η phase precipitation. The χ and η phase precipitates were created by the δ ferrite transformation due to the heat treatment.

The mentioned observations are in agreement with works of [8 - 10, 12 - 14] who observed that long term exposition of AISI 316 stainless steel led to precipitation of carbides ($M_{23}C_6$ and M_6C are the most known) and σ, χ and η intermetallic phases. Creation of σ, χ and η intermetallic phases was studied in [8 - 10].

Besides mentioned microstructural changes also the recrystallization of the microstructure was observed and increase of amount of the annealing twins was observed, Fig. 3c.

In the Fig. 4 the fatigue test results of the experimental material in the basic state and after heat treatment are shown (S-N diagram). Fatigue behavior of the tested steel was not significantly influenced by structural sensitization due to the heat treatment. In the region bellow 10^5 cycles the fatigue strength decreased due to the structural sensitization.

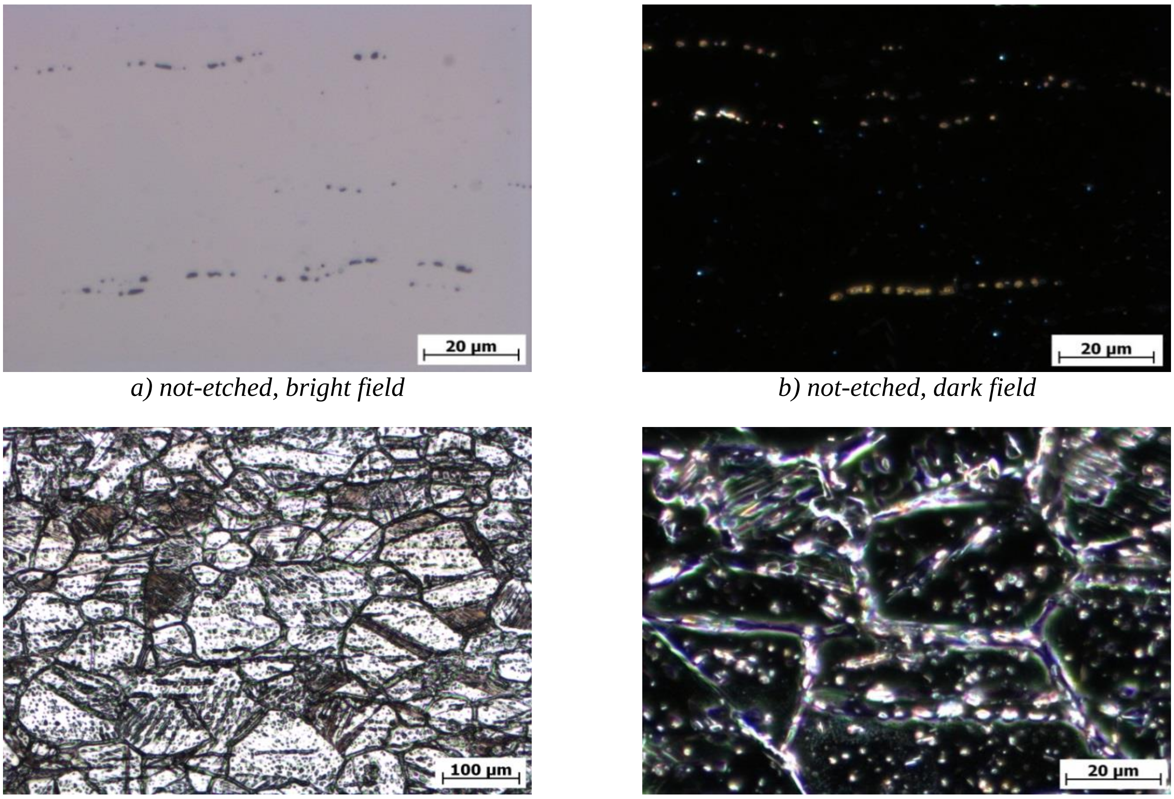


a) not-etched, bright field *b) not-etched, dark field*

c) etched, bright field *d) etched, dark field*

Fig. 2. Microstructure of AISI 316L in a basic state.

(full colour version available online)



a) not-etched, bright field *b) not-etched, dark field*

c) etched, bright field *d) etched, dark field*

Fig. 3. Microstructure of AISI 316L after structure sensitization.

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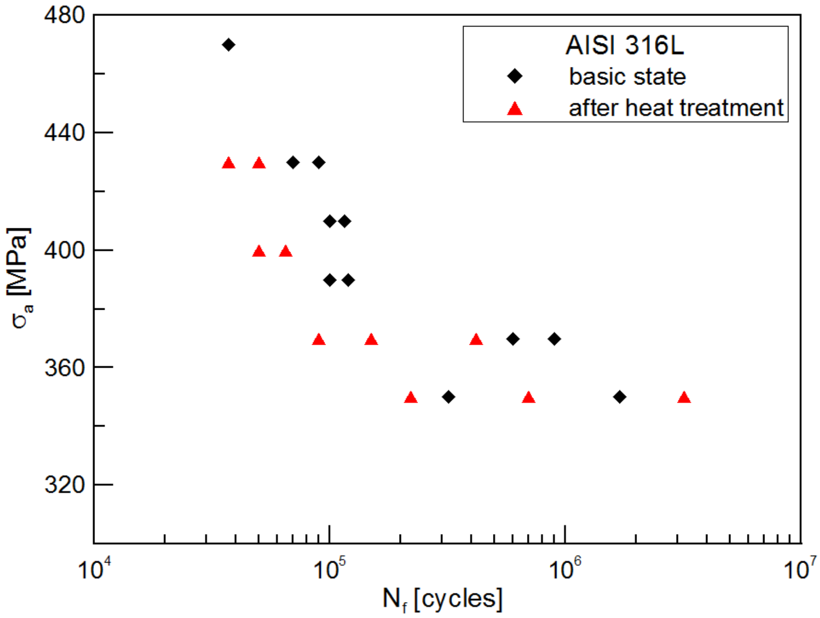

Fig. 4. Rotating bending fatigue tests results of AISI 316L in basic state and after structure sensitization.
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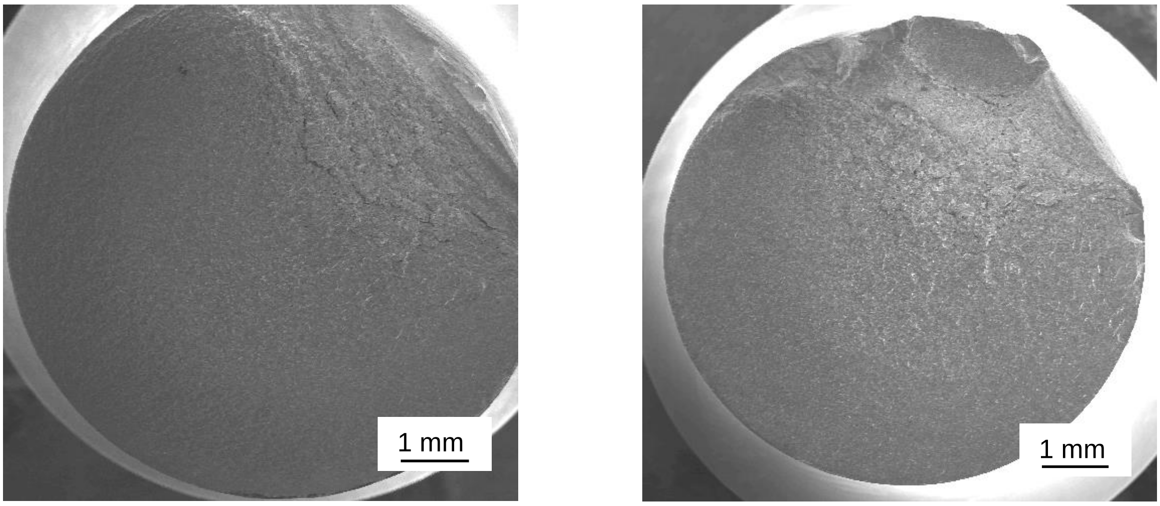


a) fracture surface of basic material, σ_a = 373 MPa, N_f= 5 015 980 cycles

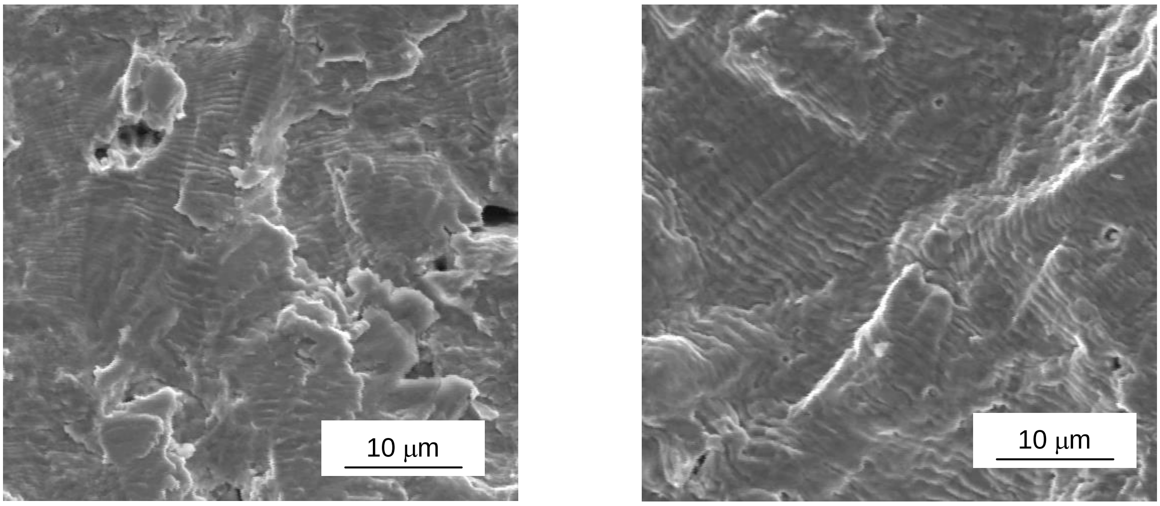
b) fracture surface of heat treated material, σ_a = 351 MPa, N_f= 744 732 cycles

Fig. 5. Fracture surfaces of tested specimens.

Below the 10^5 cycles the higher number of cycles was reached by testing bars from the basic material and the fatigue life was double when compared to the heat treated specimens. However the fatigue limit remained unchanged despite the microstructure sensitization.

Fracture surfaces of tested fatigue bars were observed with the aim to identify the fatigue crack initiation places. Fracture surfaces documented by SEM are shown in Fig. 5. In all cases the fatigue crack initiation from the surface was observed thus, no influence of the precipitated carbides on the fatigue crack initiation was observed.

The mechanism of fatigue crack growth remains also the same without any changes, see Fig. 6. In both states of the material the crack propagated by transcrystalline fatigue mechanism.



a) transcrystaline fatigue fracture of basic material, σ_a = 373 MPa, N_f = 5 015 980 cycles

b) transcrystaline fatigue fracture of heat treated material, σ_a = 351 MPa, N_f = 744 732 cycles

Fig. 6. Fracture surfaces of tested specimens - mechanism of fatigue crack growth.

4. Conclusions

Performed experiments were focused on analysis of the influence of material structural sensitization on the fatigue behavior of the AISI 316L austenitic stainless steel. Based on the obtained results following conclusions can be drowned:

- AISI 316L structural sensitization due to a long term annealing resulted in the precipitation of large amount of carbides and χ and η phase on the grain boundaries and also in the grains,

- only small decrease of the fatigue strength was observed due to AISI 316L structural sensitization and the negative effect was more significant at higher loading amplitudes,

- fatigue limit remained almost unchanged due to the structural sensitization.

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